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NATIONAL BUREAU OF STANDARDS REPORT

9996

MICROPLASTICITY I – MEASUREMENT OF SMALL MICROSTRAINS AT AMBIENT TEMPERATURE

To

Materials and Processing Branch
Naval Air Systems Command
Department of the Navy

U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

NATIONAL BUREAU OF STANDARDS

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To
Materials and Processing Branch
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U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

MICROPLASTICITY I - MEASUREMENT OF SMALL MICROSTRAINS AT AMBIENT TEMPERATURE

INTRODUCTION

This paper presents some important features observed in a study of microplasticity in metals. Investigations of microplasticity usually are concerned with very small strains of the order of 10^{-7} to 10^{-5} in materials that are stressed, generally, much below their normal yield strengths. The various microstrain measuring techniques used by investigators and the microstrain behavior of selected materials have been reviewed extensively in a very recent publication (1). Microstrain measurements generally have been made using electrical resistance gages, variable inductance transformers, Tuckerman strain gages, or capacitance gages. The capacitance gage technique was selected for the measurement of microstrains in this study primarily because it has a very high sensitivity, on the order of 10^{-7} to 10^{-8} in./in.

Microstrain measurements limited to the gage length portion of a tensile specimen was a prime requirement in the present study. In many of the previous investigations (2 to 9) in which a capacitance gage was used for the determination of microstrains introduced into a tensile specimen, the gage was usually attached to the shoulders of the specimen. The recorded strain also included the strains in the fillet sections and those portions of the reduced section outside of the gage length. Thus, these readings did not provide the more desirable measurement of the actual strain over the gage length.

As the study progressed, certain effects related to temperature changes caused by loading and unloading specimens became apparent, which seem to have been neglected in most reported studies dealing with microplasticity. These effects have now been studied quantitatively and their importance in reporting microplasticity data established by the findings in this paper.

SPECIMEN AND GAGE DESIGNS

A tensile specimen and a capacitance gage holder were specially designed to permit measurement of the actual strain over the gage length. The specimen (Fig. 1) is a modification of ribbed specimen designs previously reported (10, 11). The reduced section of 0.450 inch diameter has two 45° angle circumferential ribs of 0.05 inch height. These ribs are spaced two inches apart for attachment of the capacitance gage holders. The capacitance gage holders are designed for positioning three gages spaced 120° apart on the specimen. The gages, as installed on a specimen are shown in Fig. 2. The capacitance probes are positioned in the upper holder and micrometers fitted with small plate caps on the micrometer spindles are held in alignment with the capacitance probes by the lower holder. This micrometer arrangement facilitates positioning of the micrometer plate caps very close, normally within 0.001 inch or less, to the ends of the capacitance probes. Each holder consists of two parts with an inner groove machined to match the rib on the specimen with about 0.01 inch clearance between the holder and the reduced section of the specimen. The two halves of each holder are held together by two guide

pins with adjustable compression springs. This holder design was selected in preference to a knife edge or set screw type holder, since some slippage generally occurs with the latter types. Even though slippage might be very small, it would definitely invalidate microstrain measurements.

Any bending of the specimen during a test will be magnified greatly, as revealed by different strain readings of the three capacitance gages whose axes are 0.75 inch from the axis of the specimen. Moreover, the average strain of the specimen can be determined readily from the strain readings of the three gages. The microstrain values reported in this study are average strain values, except the strains determined within one minute after unloading of the specimen. These latter strains were obtained from readings of one gage.

TEST PROCEDURES

Preliminary tests demonstrated that reliable and reproducible strain measurements, within $\pm 1 \times 10^{-7}$, could be made with the NBS capacitance gage assembly provided special precautions were taken in the testing procedures and in maintaining precise control of the temperature of the specimen, gages, and other components of the test equipment. To achieve this it was found necessary to enclose the specimen, specimen holders and pull rods, and the capacitance gages in a thermally insulated chamber that could be maintained at a constant temperature. The temperature selected, 24.2° C, was 1 to 2° C above the thermostat-controlled temperature of the laboratory, and it was maintained to within ± 0.01° C with a proportional-power type thermistor controller. The precision variable-capacitor used in the capacitance gage bridge circuit also was enclosed

in a temperature controlled, thermally-insulated chamber, as it was found to be temperature sensitive. The coaxial wires, connectors and switches used in the measuring circuit of the capacitance gages were shielded. In addition, metal foil was placed on the inside of the thermal chamber enclosing the specimen and gages to minimize pick up of stray electrical fields.

The microplastic behavior of a specimen was determined by employing a step loading-unloading procedure, loading to successively higher peak stresses. Measurements of the residual (plastic) microstrain were repeated after complete unloading following each loading to successively higher peak stresses. Consistent measurements of the microstrain of a specimen to within $\pm$ 1 x 10^{-7} while attempting to maintain a constant low load with the hydraulic tensile machine used in these tests were not attainable; a very slight variation of only one fourth of a pound in the load on a steel specimen of 0.450 inch diameter varies the elastic extension of a 2 inch gage length by approximately 1 x 10^{-7} inch.

EXPERIMENTAL RESULTS

Initial Tests on Type 4340 Steel. Preliminary results were obtained on a tensile specimen machined from a bar of normalized 4340 steel that had been heated at 900° C (1650° F) for one hour and air cooled. The 4340 steel was vacuum melted and had the following chemical composition in percent by weight: carbon 0.40, manganese 0.68, phosphorus < 0.01, sulphur < 0.01, silicon 0.33, nickel 1.80, chromium 0.79, molybdenum 0.24, balance iron.

The microstructure of the normalized steel consisted mainly of lower bainite and martensite. Its yield strength (0.2% offset) was determined to be 151 ksi.

The initial test data revealed that the observed microstrain readings depended upon the interval between complete unloading of the specimen and the strain readings. Consistent strain readings at zero load generally were obtainable only after a waiting period of about 10 minutes, as a small continuously decreasing strain (contraction of the specimen) with increase in time was observed during this period. This characteristic is illustrated in Fig. 3 for three tests on a single specimen. The specimen was loaded to stresses of 51, 52 and 53 ksi, representing about one-third of the yield strength. The specimen was loaded and unloaded at a rate of approximately 10 ksi/min. In the two tests represented by curves 1 and 2, the first strain measurements were obtained 50 and 30 seconds, respectively, after complete unloading of the specimen, whereas, in the test represented by curve 3, (Fig. 3 part c) the first strain measurement was obtained 10 seconds after unloading of the specimen. As an appreciable decrease in strain apparently had occurred prior to the first strain measurements in the two tests represented by curves 1 and 2, the curves were normalized with curve 3 at the 1 minute value and the normalized curves are shown in part c of Fig. 3.

The observed total decreases in strain with time suggested to the authors that a decrease of about 0.06° C in the temperature of the specimen probably had occurred during the time involved in each of the three series of strain readings. This suggested temperature change was

based upon a value of 11.3×10^{-6} in/in/°C for the coefficient of linear thermal expansion of 4340 steel. Some cooling of the specimen during loading within its elastic range and heating during unloading is to be expected from thermodynamic considerations (12). Elastic extension of a material with a positive coefficient of linear thermal expansion under adiabatic loading conditions should decrease the temperature of the material. Conversely, elastic contraction that occurs during elastic unloading of the material should increase its temperature.

Temperature Changes of AISI 4340 Steel Specimen Accompanying Loading and Unloading within its Elastic Range. A literature search did not reveal any data on the specific temperature change of steel during loading and unloading within its elastic range. Consequently, a series of tests were conducted on the normalized 4340 steel specimen in which its temperature changes during loading, unloading, and the subsequent zero load condition were measured. A 22 gage Chromel-Alumel thermocouple was taped tightly to the surface of the specimen at the midpoint of the gage-length section and a zero suppression, multi-range milli-micro-voltmeter (ranges varying from 0 - 0.1 μV full scale, to 0 - 100 μV full scale) was used to determine the temperature changes.

In the first series of tests the specimen was loaded within its elastic range at selected loading rates to a stress of 50 ksi. This stress was maintained until the specimen regained its initial temperature. The specimen then was unloaded at the same rate. Representative data are presented in Fig. 4.

The data obtained in a test at a relatively slow loading rate of 3.1 ksi/min. are shown in part a. Special attention should be directed to the temperature decrease observed during loading and again to the temperature increase during unloading. The temperature changes were appreciable, even though testing at this slow loading rate did not approach adiabatic conditions, and there was a considerable heat flow into and out of the specimen through its holders and its environment.

Data obtained in a test at a higher loading rate of 19 ksi/min. are shown in Fig. 4, part b. The heat transfer was less and consequently the temperature changes were about three times greater than those observed in the test conducted at the loading rate of 3.1 ksi/min.

Larger temperature changes were observed (Fig. 4, part c) in the test at a high loading rate of 137 ksi/min. This test closely approached an adiabatic test condition with very little heat transfer. A slight time lag was observed in the decrease of the surface temperature of the specimen on loading. This is indicated by the occurrence of a minimum surface temperature several seconds after application of the maximum loading stress of 50 ksi. Attention also should be directed to the time (10 to 20 minutes) that was required for the specimen to regain its initial temperature, both after loading to and holding at 50 ksi, and after complete unloading. Similar data (not shown) were obtained in additional tests at other loading rates.

A second series of tests was conducted in which the specimen was stressed within its elastic range at selected loading rates to 50 ksi and then immediately unloaded at the same rate. (Loading followed by immediate unloading is the method usually employed in microplasticity

studies.) Representative data are presented in Fig. 5.

At a slow loading rate of 4.7 ksi/min. (Fig. 5, a) heat transfer into the cooled specimen was sufficient for it to regain its initial temperature value early in the unloading stage at about 44 ksi. Further heating of the specimen generally occurred during the remainder of the unloading stage producing a temperature at 12.5 ksi that was nearly 0.05° C above its initial temperature. This was followed by slow cooling during the further unloading of the specimen; the heat transferred out of the specimen to its holders and environment was greater than the heat generated in the specimen by its additional elastic contraction.

The temperature changes of the specimen during a test at a loading rate of 19 ksi/min. (Fig. 5,b) were greater than those observed in the test at the slower loading rate. However, the increase in temperature of the specimen above its initial value at complete unloading was much less than the temperature decrease on loading to the 50 ksi stress.

The temperature changes observed in a test at a very high loading rate of 380 ksi/min. are presented in Fig. 5c. This test closely approaches adiabatic conditions with very little heat transfer during the loading and unloading. Moreover, the change in the surface temperature of the specimen apparently lagged behind that in the interior, and this resulted in the maximum decrease in the surface temperature occurring at the 49 ksi stress during unloading. At the instant of complete unloading of the specimen its surface temperature was slightly below its initial value and it continued to increase for about 40 seconds,

attaining a value slightly above its initial temperature. This increase in the surface temperature after complete unloading can be attributed to the temperature gradient existing in the cross section of the specimen. Moreover, even though the duration of the loading and unloading stages was very short (about 20 seconds) there was a very small transfer of heat into the specimen since its temperature during loading and unloading was below the initial temperature. The observed increase in temperature above the initial temperature can be attributed to this heat transfer.

The time required for the specimen to attain temperature equilibrium (to regain its initial temperature) after unloading in these tests (Fig. 5) and in similar tests (not shown) at other loading rates was generally about 10 minutes.

The data presented in Figs. 4 and 5 and those obtained in similar tests are summarized in Fig. 6. These graphs depict the influence of the loading rate on the maximum changes in temperature of the specimen during loading to 50 ksi stress followed by either immediate or delayed unloading.

The two curves in Fig. 6 for tests in which unloading was delayed until the specimen had regained its initial temperature reveal that the decrease in temperature of the specimen by the loading was numerically equal to the increase in the temperature of the specimen by unloading at the same rate. These curves also illustrate the dependence of the maximum temperature changes on the approachability to adiabatic test conditions. Adiabatic conditions apparently were approached closely at loading rates of about 100 ksi/min. and higher, as indicated by the nearly constant values of the maximum temperature changes at these rates.

The two curves in Fig. 6 for the tests in which the specimen was unloaded immediately after loading to 50 ksi stress also illustrate indirectly the approachability to adiabatic test conditions. If the test conditions were completely adiabatic the temperature of the specimen after unloading should be the same as its initial value. Thus, the increases in temperature above the initial temperature of the specimen that were observed following the complete unloading is a relative indication of the heat transfer into the specimen during the test.

Temperature Changes of Invar Accompanying Loading and Unloading Within its Elastic Range. The degree of cooling and heating of a specimen during loading and unloading within its elastic range should depend upon its coefficient of linear thermal expansion. For conformation of this characteristic a test was conducted at 24.2°C on a specimen of annealed Invar with a very low positive coefficient of linear thermal expansion at ambient temperatures (approximately 1×10^{-6} in/in/°C which is less than one-tenth that of the 4340 steel). This specimen was machined from a sample of Invar that had been given the prescribed annealing treatment of 830° C (1525° F) for one hour followed by quenching in water. Its yield strength (0.2 percent offset) was determined to be 43.1 ksi. The specimen was prestressed in microplasticity tests to 15 ksi with a total microstrain of 15×10^{-6}. This prior treatment was selected to minimize any plastic microstraining of the specimen during subsequent tests in which temperature changes were measured. The temperature changes observed during loading and unloading of the specimen within its elastic range at a rate of approximately 10 ksi/min. to stresses of 5, 10, and 15 ksi are presented in Fig. 7. A very slight, although

significant, cooling occurred during loading of the specimen and heating during unloading. These temperature changes conformed to expected values. For example, when stressed at 15 ksi the elastic strain of the Invar specimen which has a modulus of elasticity about two-thirds that of the 4340 steel, should be about one-half that of the 4340 steel specimen stressed to 50 ksi. Furthermore, with the coefficient of linear thermal expansion of the Invar specimen equal to approximately one-tenth that of the 4340 steel specimen, its observed temperature change of -0.007° C at the 15 ksi stress (Fig. 7) should be, and was, approximately one-twentieth of the temperature change of -0.13° C observed for the 4340 specimen stressed to 50 ksi at a similar elastic strain rate. It should be noted that to obtain similar elastic strain rates, loading of the Invar specimen at 10 ksi/min. corresponds to loading of the 4340 specimen at about 15 ksi/min.

Temperature Changes of Normalized 4340 Steel Specimen during Loading into Microplastic Deformation Range and Unloading. Measurements were made of the changes in temperature of the normalized 4340 steel specimen accompanying the loading into the microplastic deformation range, unloading, and the subsequent zero load condition. The specimen prior to this test had been stressed in step loading-unloading microplasticity tests to a peak stress of 110 ksi with a total residual microstrain of 830×10^{-6}. The temperature changes observed in a test conducted at a loading rate of 18 ksi/min. to a stress of 129 ksi, followed by immediate unloading at the same rate are presented in Fig. 8.

The temperature of the specimen decreased during the elastic loading to a stress of approximately 110 ksi. As the stress increased from 110 to 129 ksi a plastic strain of 367×10^{-6} occurred, accompanied by heating of the specimen. The heating effect due to the plastic deformation on increasing stress from 110 to 129 ksi was much greater than the cooling effect due to the accompanying increase in elastic strain. The temperature continued to increase during unloading, reaching a maximum of 0.2° C above the initial temperature of the specimen. The time required for the specimen to cool to its initial temperature after unloading was about 30 minutes.

The above test and the previously reported tests in which the temperature changes of specimens of normalized 4340 steel and annealed Invar were monitored, clearly demonstrate that reliable plastic microstrains usually cannot be readily determined from readings taken immediately or very soon after complete unloading of the specimen, due to the thermal strains that occur during the interval required for the specimen to regain its initial temperature. These thermal strains can be minimized, but not completely eliminated, by step loading and unloading (1) at very low loading rates, probably requiring an hour or more for the loading-unloading step or (2) at very high loading rates approaching adiabatic test conditions. Thus, any reported data obtained in microplasticity tests at ambient temperature in which the microstrain readings were taken immediately or very quickly after unloading of the specimen may be of questionable value due to the inclusion of unmeasured thermal strains. Moreover, any microstrain readings during the loading-unloading stage, such as those obtained through automatic recording of tests at ambient temperature, will include effects of thermal contractions and

expansions along with the elastic strains as well as plastic strains if the specimen had been stressed into the microplastic deformation range.

SUMMARY

A tensile specimen and a capacitance gage assembly were designed for determination of small residual microstrains to a sensitivity of $\pm 1 \times 10^{-7}$ over a 2-inch gage-length section.

A study was made of the microplastic behavior of a specimen of normalized 4340 steel, employing a step loading-unloading procedure, loading to successively higher stresses. The residual microstrains were determined at zero stress after each unloading of the specimen. In order to obtain consistent and reliable measurements of residual microstrains to within $\pm 1 \times 10^{-7}$, special test procedures were required; these and some observed characteristics follow:

(1) The specimen and capacitance gage assembly must be maintained at a constant temperature to within $\pm 0.01^{\circ}$ C prior to loading the specimen and during the gage readings following the complete unloading of the specimen. This required the enclosing of the specimen, specimen holders and pull rods, and the capacitance gage assembly in an insulated thermal chamber controlled to within $\pm 0.01^{\circ}$ C.

(2) Satisfactory measurements of residual microstrains were not plausible while maintaining a small load on the specimen with the tensile machine. For example, a slight variation of only 4 ounces in the load on the 0.450 inch diameter specimen of 4340 steel changes the elastic extension of a 2-inch gage-length section by approximately 1×10^{-7} inch.

(3) Small changes in temperature that accompany the loading and unloading of the specimen must be taken into account. The 4340 steel specimen with a positive coefficient of linear thermal expansion undergoes significant cooling during loading within its elastic range and heating during unloading.

Appreciable heating also occurs during microplastic deformation, even though the microstrain of the specimen is very small. Thus, the specimen at or immediately after unloading is not at its initial temperature, i.e., the controlled temperature of the thermal chamber. An interval of 10 or more minutes was generally required for the specimen to regain its initial temperature.

Microstrain measurements determined during loading or unloading of specimens will include thermal strains due to temperature changes, along with the elastic strain and any plastic strain of the specimen. Moreover, microstrain measurements taken immediately after unloading of the specimen will include some thermal strains. An interval of 10 or more minutes following unloading usually was required prior to taking microstrain readings to eliminate thermal strains and provide reliable residual microstrains; when the plastic microstrain during the test was very large, such as 367×10^{-6} or greater, a waiting period of 30 or more minutes was required.

Thermodynamical theory indicates that the temperature changes during loading within the elastic range and unloading should be relatively small for a material which has a low coefficient of linear thermal expansion. To confirm this behavior temperature changes were monitored in a test conducted at 24.2° C on a specimen of annealed Invar which had a very

low coefficient of linear thermal expansion at ambient temperatures. The temperature changes of the specimen were very small, less than 0.01° C, during loading and unloading within its elastic range to stresses of 5, 10 and 15 ksi. The specimen regained its initial temperature within one minute after unloading. The accompanying thermal strains of the specimen were too small to be readily measured.

ACKNOWLEDGMENTS

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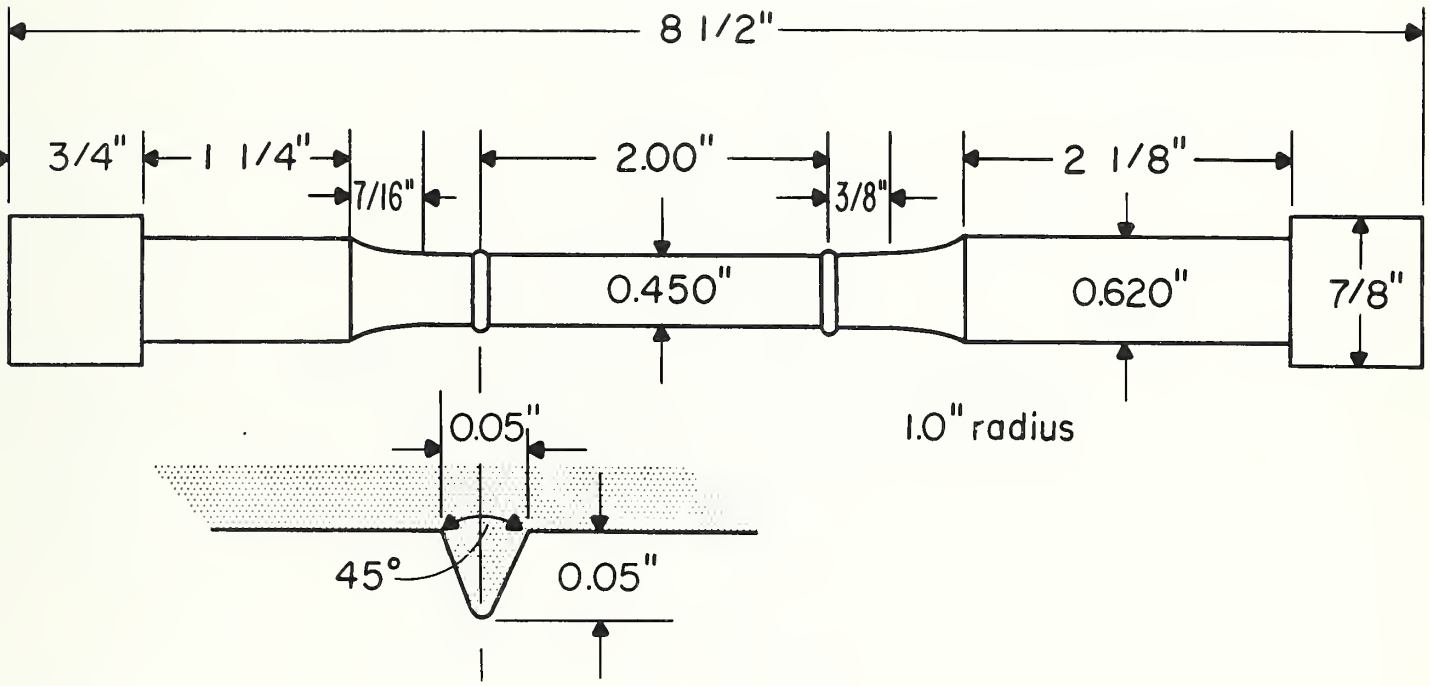

FIG. 1. MICROPLASTICITY SPECIMEN

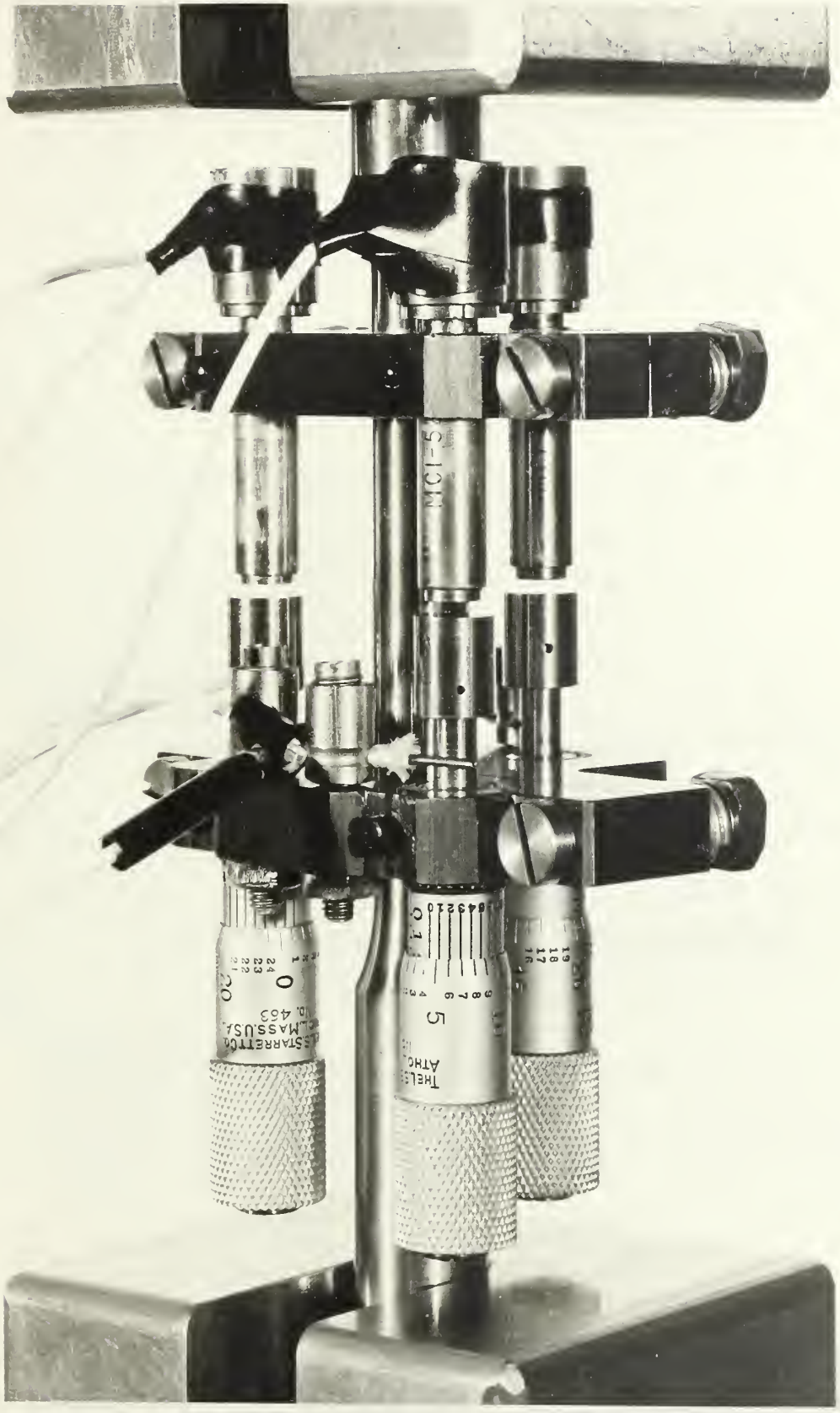

FIG. 2 CAPACITANCE GAGES INSTALLED ON SPECIMEN

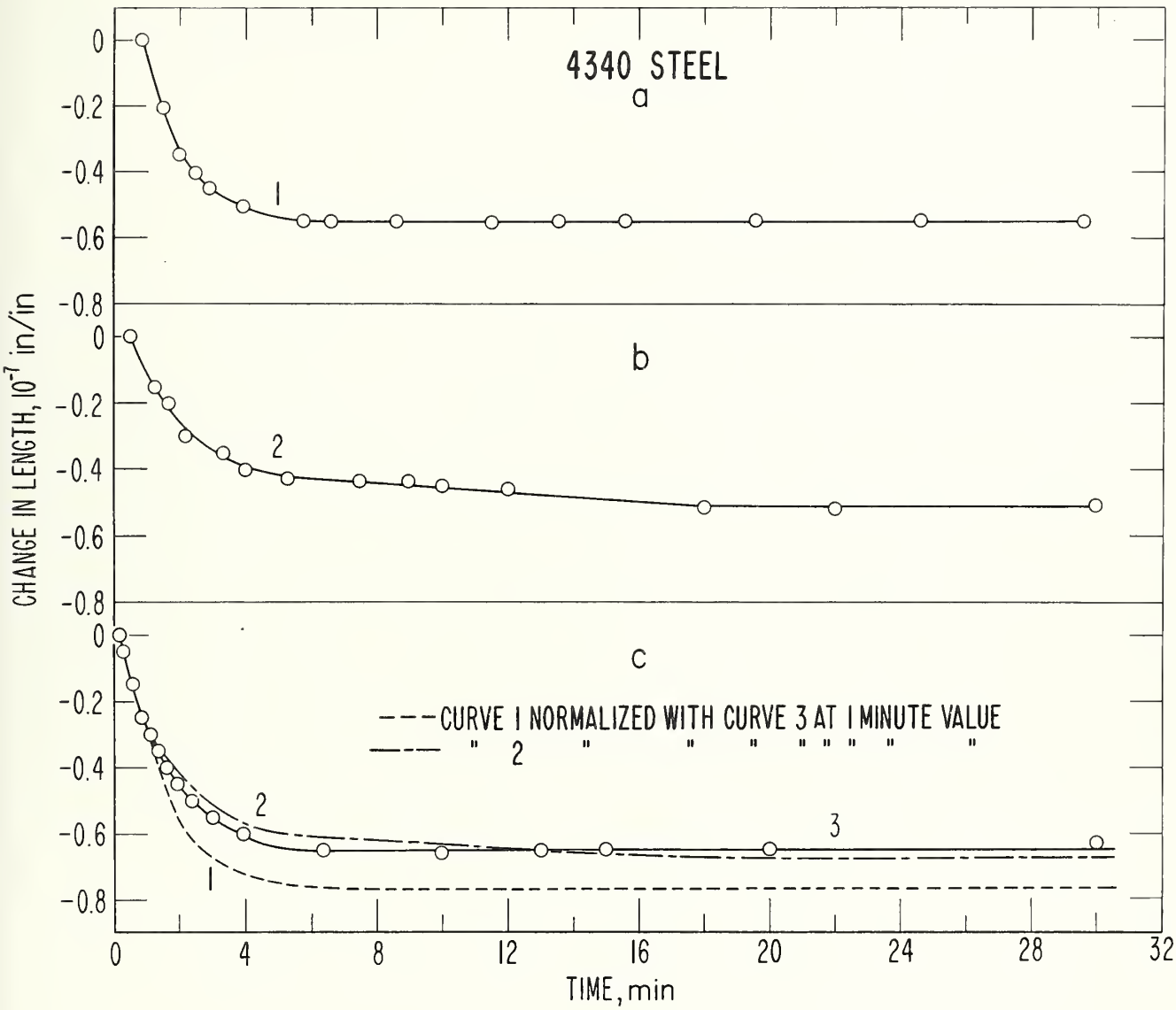

FIG. 3. Decrease in total strain with time after complete unloading of normalized 4340 steel specimen. Specimen loaded within its elastic range at approximately 9 ksi/min (1 MN/m^2/sec) and immediately unloaded at same rate. (a) Stressed to 51 ksi (352 MN/m^2); (b) Stressed to 52 ksi (359 MN/m^2); and (c) stressed to 53 ksi (365 MN/m^2).

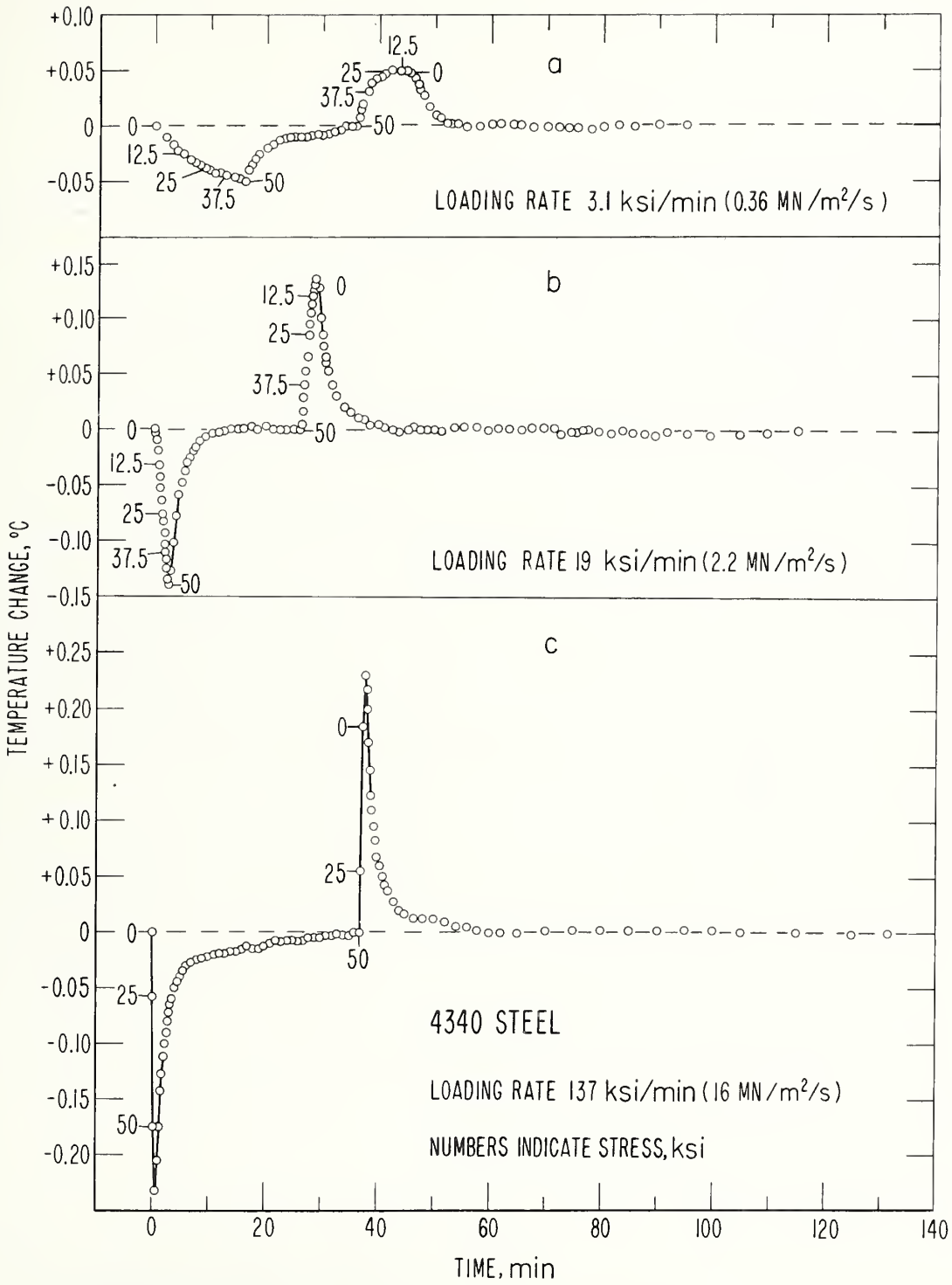

FIG. 4. Temperature changes or normalized 4340 steel specimen during test. Specimen loaded within its elastic range at selected rates to 50 ksi (345 MN/m^2) with delayed unloading at same rate.

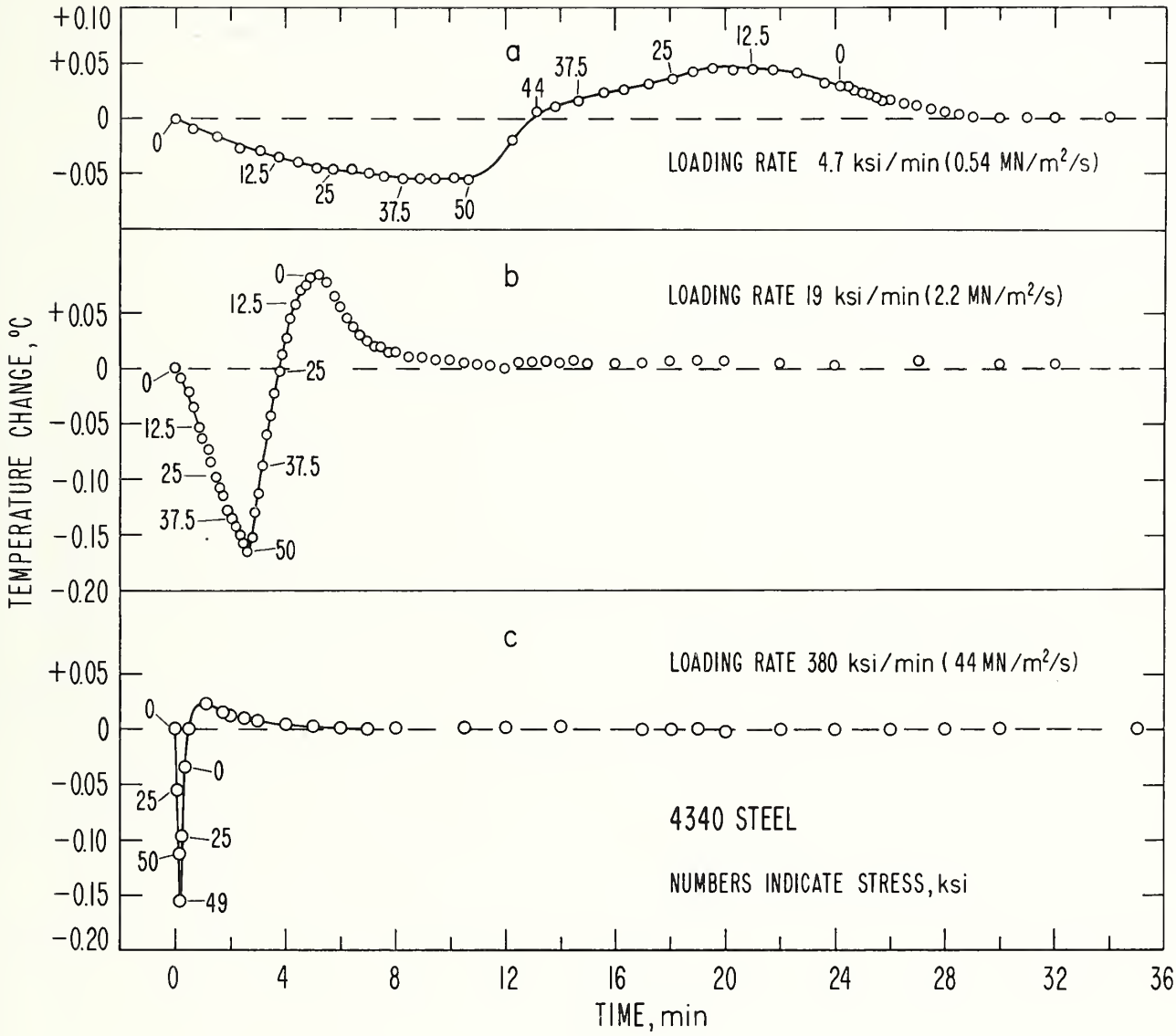

FIG. 5. Temperature changes of normalized 4340 steel specimen during test. Specimen loaded within its elastic range at selected rates to 50 ksi (345 MN/m^2) and immediately unloaded at same rate.

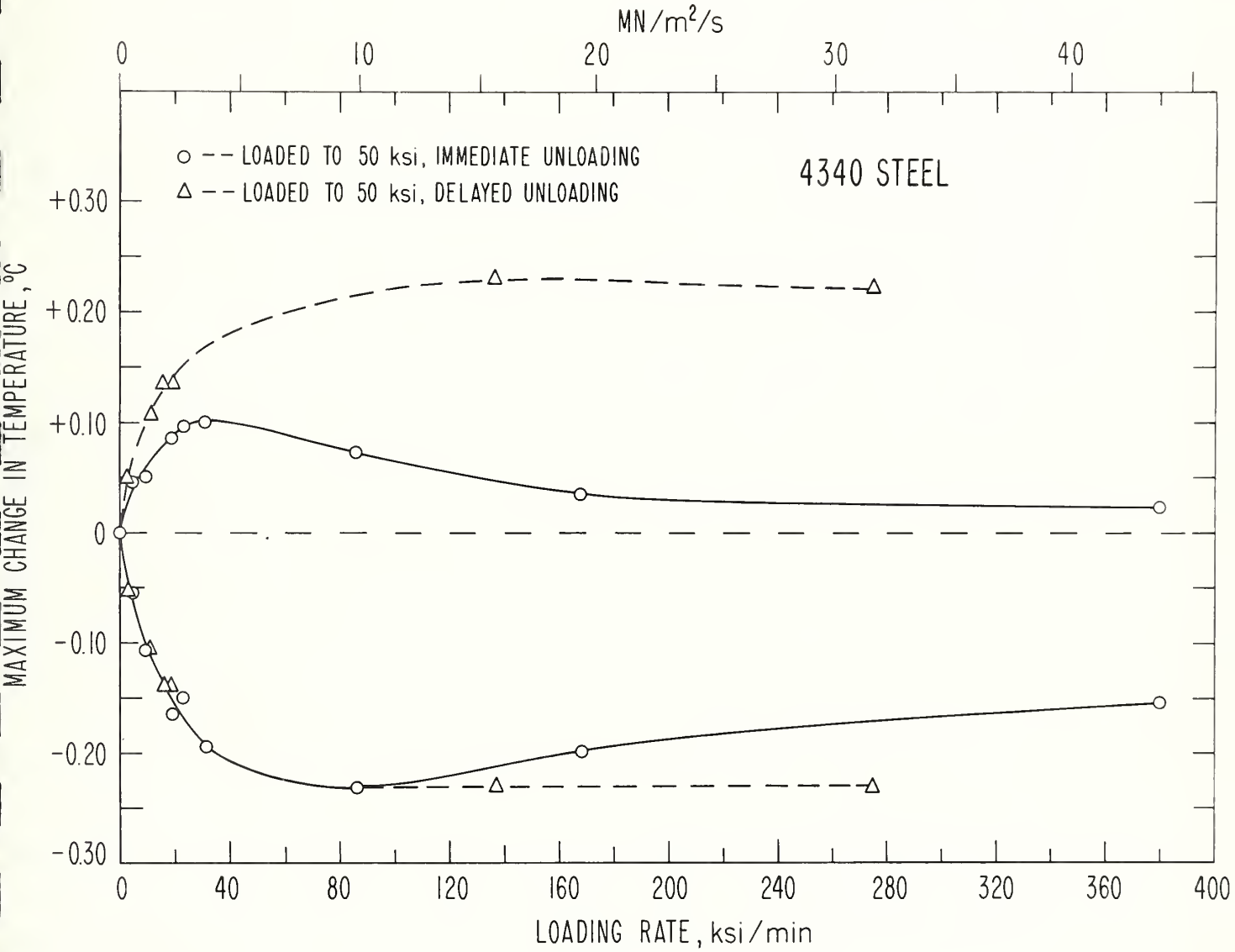

FIG. 6. Effect of loading rate on maximum temperature changes of normalized 4340 steel specimen stressed to 50 ksi (345 MN/m^2) and unloaded.

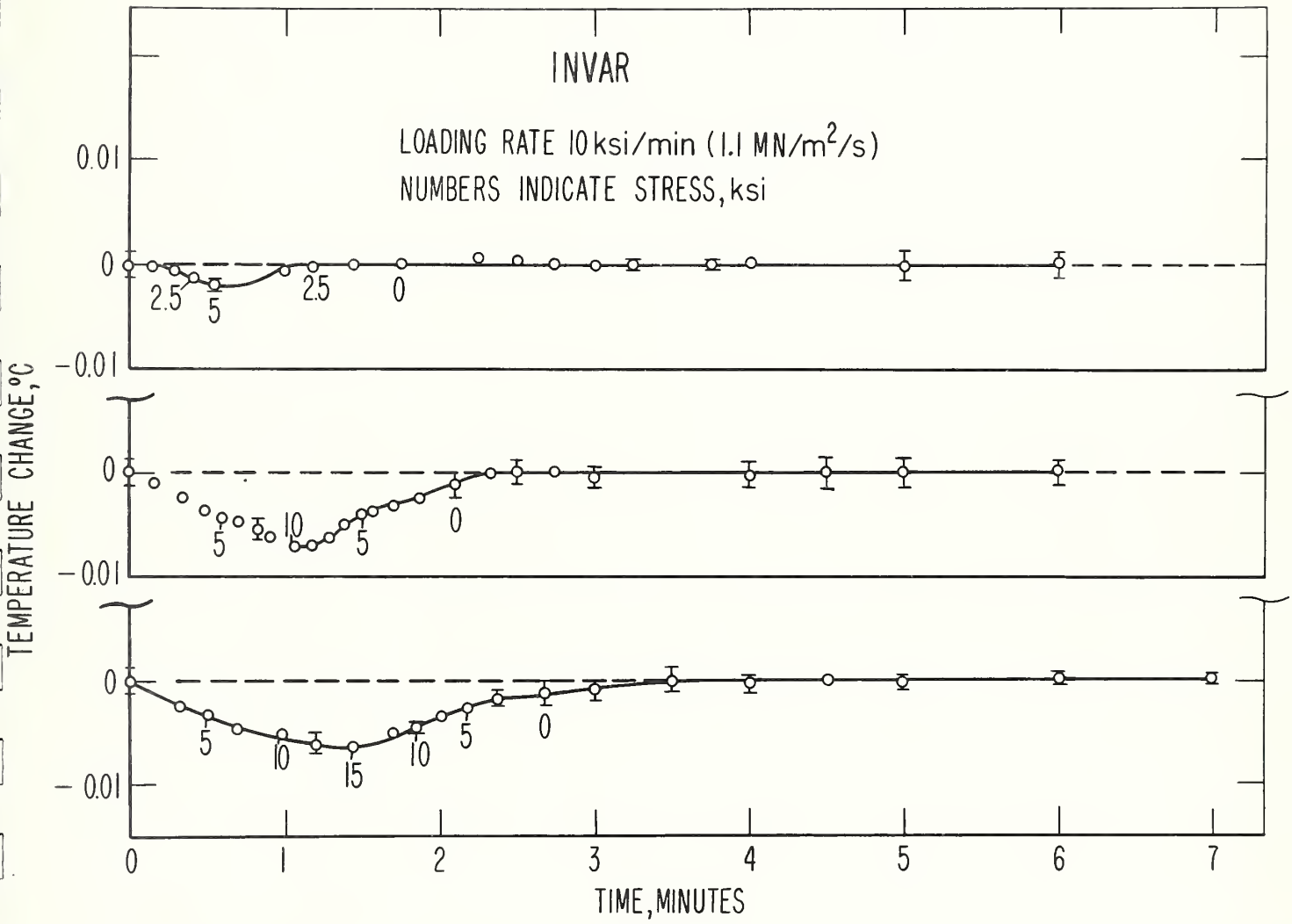

FIG. 7. Temperature changes of annealed Invar specimen during test. Specimen loaded at a rate of 10 ksi/min (1.1 MN/m^2/s) to selected stresses within its elastic range and immediately unloaded at the same rate.

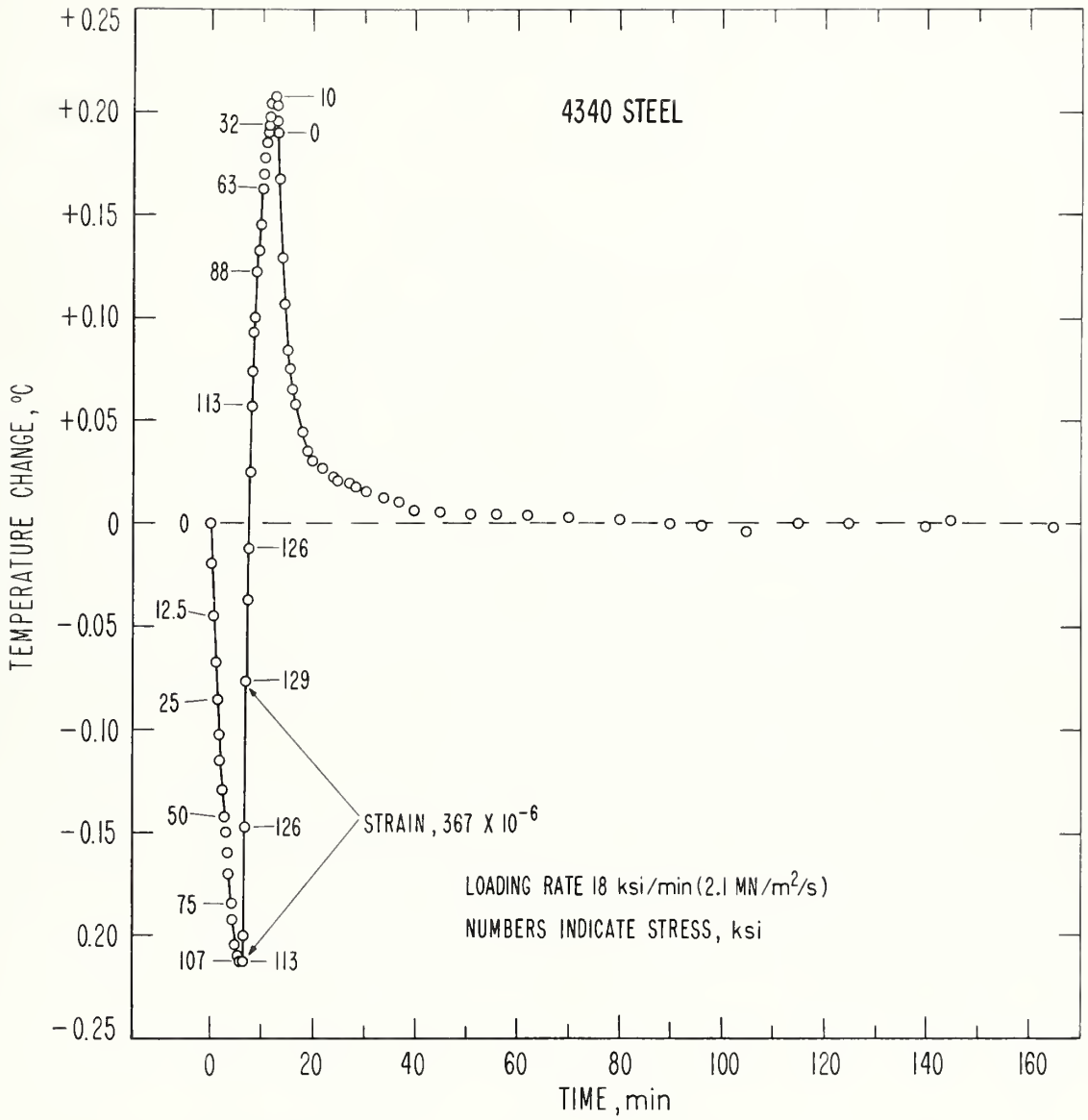

FIG. 8. Temperature changes of normalized 4340 steel specimen during loading into its microplastic range. Specimen loaded at a rate of 19 ksi/min (2.2 $MN/m^2/s$) to 129 ksi (890 MN/m^2) and immediately unloaded at the same rate.

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